2632

Possible Inclusion of Artifacts in Flavors Recovered Directly from Dried Whole Milk

F. E. KURTZ

Eastern Utilization Research and Development Division, USDA, Washington, D. C.

Abstract

To determine possible sources of artifacts arising in collecting volatile flavors from dehydrated dairy products, a study was made of contributions derived from the components of a high-vacuum collection procedure.

Volatiles from 115 cm^2 of silicone high vacuum grease, Apiezon-N grease, or Teflon gaskets, collected on a liquid nitrogen-cooled condenser by holding each material for 3 hr at 25 C and 1-μ pressure, when added to 200 ml of milk, were undetected by a trained taste panel. Under the same conditions, volatiles from silicone, neoprene, and buna-N gaskets and O-rings were easily detected, lowering the flavor score of the milk pronouncedly. Viton O-rings produced a borderline effect: 115 cm^2 of material lowered the flavor score of milk slightly, while the volatiles from 57 cm^2 could not be detected.

The recovery process had a generally deleterious effect on dried whole milk. The magnitude of this effect varied from barely detectable with some powders to quite definite with one powder. This effect was shown to depend on the hydration of the powder, being absent when nonhydrated powders were treated otherwise identically.

Two pertinent questions regarding any procedure for isolating flavor compounds from food products concern the completeness of recovery and the extent to which foreign flavors may be introduced by the recovery procedure. With respect to a procedure which has been described for recovering off-flavors directly from dried whole milk, data on the recovery of added ketones (3) furnish information relevant to the first question; although it should be emphasized that flavor compounds might be recovered with more difficulty than expected if their volatility were reduced by, for example, complexing with other constituents. Relevant to the second question, this paper presents data regarding the possibility of recovered flavors being contaminated with foreign off-flavor compounds, either originating in the apparatus or produced in the milk powder itself, as a result of the recovery process.

Materials and Procedures[1]

Lubricants and gaskets. Silicone high vacuum grease, Apiezon-N grease, silicone, neoprene, and Teflon gaskets, and buna-N and Viton O-rings were used as purchased from supply houses.

Foam-dried whole milks. Powder C was foam-vacuum-dried (5), canned in nitrogen, and held at −18 C until used. The fat in the untreated powder was present as 51% free fat, as determined by a previously described method (3). Powders A, B, D, E, and F were all foam spray-dried (2), canned in nitrogen cotaining 5% hydrogen with a palladium catalyst (1), and held at 4 C until used. The free fat content of the untreated powders was: A. 30%; B, 18%; D, 22%; E, 11%; and F, 17%.

Recovery and evalution of condensed volatiles. Volatile materials were collected in an apparatus previously described (3). Each lubricant was spread on microscope slides to an area of approximately 115 cm^2 and placed in a five-liter flask, attached directly to a U-tube containing stainless steel balls. Volatiles were collected for 3 hr at a pressure of 1 μ, with the flask containing the sample held at 25 C and the U-tube cooled with liquid nitrogen. At the end of the

[1] Reference to certain products or companies does not imply an endorsement by the Department over others not mentioned.

TABLE 1

Recovery of off flavors from lubricants and gasket materials

Material tested	Panel ratings: Milk	Panel ratings: Milk + volatiles	Panel ratings: Average lowering of score
Silicone high-vacuum grease	37.3	37.3	0.0
Apiezon-N grease	37.3	37.3	0.0
Silicone gaskets	38.1	34.2	3.9
Neoprene gaskets	38.1	33.8	4.3
Teflon gaskets	37.3	37.3	0.0
Buna-N O-rings	36.7	33.0	3.7
Viton O-rings	37.3	36.3	1.0

collection period, the system was brought to atmospheric pressure with nitrogen, the U-tube removed from the cold bath, nearly filled with milk, and warmed in 25 C water until the milk remained unfrozen. In all, a total of 200 ml of milk was used to rinse out the U-tube. The sample of milk containing condensed volatiles, along with a control milk, was held overnight at 4 C, then evaluated over a ten-point range, as previously described (4) by an expert-type taste panel of five judges. Volatiles from O-rings and gaskets having, likewise, a surface area of 115 cm^2, were collected and treated in the same manner.

Dried whole milks. For the data of Table 2, milk powders were hydrated in vacuum and volatiles condensed in the well of a manifold, as previously described (3). Both the U-tube and manifold were rinsed with distilled water, which was then used to reconstitute milk from the residual powder. For the data of Table 3, part of each powder sample was hydrated in vacuum without subsequent heating and part heated in vacuum without prior hydration. As before, any condensed volatiles present were collected and combined with the residual powder during reconstitution.

TABLE 2

Effect of direct recovery procedure on flavor score of dried whole milks

Milk powder	Free fat[a]	Heat treatment	Effect on flavor score[b]
A	97	40 C, 3 hr	−0.8
B	84	40 C, 3 hr	−1.0
C	80	40 C, 3 hr	−0.7
D	47	50 C, 3 hr	−0.7, −1.0
D	72	50 C, 3 hr	−1.3, −1.7
E	95	50 C, 3 hr	−1.9, −2.0
E	82	50 C, 3 hr	−1.8, −2.0

[a] In vacuum-hydrated powder.
[b] Compared with control powder reconstituted after approximately 30 min of exposure to air.

Data in Table 4 were obtained with powders which were hydrated by spraying a thin layer of powder in an open tray (while resting on the pan of a balance) with a mist of water until its weight had increased the desired amount. The moistened powder was transferred to a flask closed with a glass stopper, and the contents were thoroughly mixed by shaking. With occasional further shaking the powder was held 30 min, to allow equilibration of the moisture throughout the powder. At this time, 50 to 60 min after initial exposure to the air, portions of the hydrated powder and also of the control powder were reconstituted and held at 4 C pending evaluation by the taste panel. The remainder of the hydrated powder and, in certain instances as indicated in Table 4 a portion of the control powder, were held in air in glass-stoppered flasks at 25 C for a total of 24 hr before reconstituting.

All flavor evaluations of milks from Powders A, B, and C were made with a panel of five judges, and those from Powders D, E, and F with a panel of ten judges.

Results and Discussion

Lubricants and gaskets. The surface area of 115 cm^2 for exposing lubricants and gaskets was chosen with the thought that this was well beyond that normally present in the author's collection system. Therefore, any materials not giving detectable off flavors under these conditions would, likewise, not do so under normal conditions. However, materials giving off flavors under these exaggerated conditions might or might not do so under normal conditions, but could be considered possible sources of interference.

Volatiles condensed from silicone high vacuum grease, Apiezon-N grease, and Teflon had no

TABLE 3

Comparative effect of heating nonhydrated powders in vacuum and of vacuum hydration without subsequent heating on flavor score of dried whole milks

Milk powder	Heat treatment[a]	Free fat[b]	Flavor score: Control	Effect of heating only[c]	Effect of hydrating only[c]
A	40 C, 3 hr	97	35.6	−0.4	−1.2
B	40 C, 3 hr	84	35.8	+0.2	−0.5
C	40 C, 3 hr	80	36.2	−0.2	−0.9
D	50 C, 3 hr	91	35.3	0.0, −0.2	−0.6, −0.7
E	50 C, 3 hr	87	35.8	+0.2, +0.5	−1.2, −1.4

[a] Of nonhydrated powder.
[b] In hydrated powder.
[c] Compared with control powder reconstituted after 30 min of exposure to air.

TABLE 4

Effect of air hydration on flavor score of dried whole milks

Milk powder	Moisture[a]	Free fat		Flavor score			
				Control		Hydrated sample	
	—(%)—	—(%)—					
		(1)	(2)	(3)	(4)	(5)	(6)
D	8.1	18	30	35.4	....	−0.4	−0.2
D	9.4	33	92	35.3	+0.5	−0.1, −0.7	−1.7, −1.9
D	10.6	87	87	36.0	....	−1.0	−1.7
F	8.2	21	21	35.6	+0.1	−0.9, −0.9	−0.3, −0.4
F	9.0	21	21	35.6	+0.1	−1.0, −1.0	−1.2, −1.3
F	9.6	32	87	35.0	....	−0.6	−0.6

[a] In hydrated powder.

(1) In sample of hydrated powder taken just before reconstitution, at approximately 1 hr after initial exposure to air and 30 min after completion of hydration.

(2) In sample of hydrated powder taken just before reconstitution, after holding 24 hr in air.

(3) Control powder reconstituted after holding approximately 1 hr in air.

(4) Score, relative to value under (3), of control powder reconstituted after holding 24 hr in air.

(5) Score, relative to value under (3), of hydrated powder reconstituted after 1 hr of air exposure.

(6) Score, relative to value under (3), of hydrated powder reconstituted after holding 24 hr in air.

effect on the flavor of milk (Table 1). This was not only true for average panel scores but was true for each flavor judgment, as no judge at any time could detect any difference between the milk control and the milk with added volatiles. This is of interest, in that each of the lubricants has a typical, easily detected odor.

In contrast to Teflon and the lubricants, each of the rubbers was a source of off flavors. Silicone, neoprene, and buna-N produced large effects and every judge easily detected the presence of volatiles from each of these rubbers. Viton was borderline in its effect, as one judge could not detect its volatiles. Additional experiments, not included in Table 1, showed that when the surface area of Viton was reduced to 57 cm^2 no judge was able to detect its volatiles.

On the basis of these data, and considering the known resistance of Viton to both heat and unusually low temperatures, this material has been used in all subsequent experiments requiring rubber gaskets or O-rings. Currently, all Viton O-rings and gaskets are pretreated by overnight heating in a vacuum at 250 C, a procedure shown to be effective in removing volatiles from silicone rubber septums (7).

Dried whole milks. In Tables 2-4, all values on a horizontal line were determined in the same session of a taste panel—values on different lines represent judgments made at different sessions. It is seen (Table 2) that there is a general lowering of flavor score resulting from the recovery process, but that the magnitude of this effect varies from powder to powder.

In an attempt to determine what aspect of the recovery procedure was responsible for this lowering of score, nonhydrated powders heated in vacuum were compared with powders hydrated in vacuum, but without subsequent heating. It is seen from Table 3 that heating alone has little or no effect on the flavor score, but that hydration, even without subsequent heating, is responsible for production of off flavors.

While vacuum hydration avoids prolonged exposure to oxygen, air hydration, as described in the experimental section, permits one to follow more closely the course of the powder hydration and associated phenomena. The series of experiments involving air hydration (Table 4) were conducted with the hope of gaining a greater insight into the contribution of moisture content, freeing of the fat, and holding time on loss of flavor score.

Results (Table 4) indicate no flavor deterioration from holding either powder, while nonhydrated, in air for 24 hr compared to 1 hr. This flavor stability, however, is by no means typical of all dried whole milks (6). Powder D showed only a minor loss of flavor score from hydrating to 8.1% moisture; at higher moisture contents its initial loss of flavor score was greater and increased still further on holding. In contrast, Powder F showed no increased loss of flavor score from holding beyond 1 hr.

The data gave no indication that flavor changes are associated with the actual physical changes involved in freeing the fat. In one instance the fat in Powder F was mostly freed between the 1-hr and the 24-hr reconstitutions, and flavor scores for both samples were identical. Under similar circumstances, Powder D showed a lower flavor score for the 24-hr sample, but this was also true of another sample of D, in which the fat was freed before the 1-hr reconstitution.

Concerning the effect of moisture level on the extent of flavor deterioration, within the range studied there is no correlation discernible to the author.

Conclusions based on small differences would not be justified, as Liming (4) has reported a mean of 0.26 and a range of 0.10 to 0.39 for the monthly average difference between scores assigned to duplicate samples by the Dairy Products Laboratory taste panel. It seems clear, however, that while different powders varied in flavor stability toward hydration, moisture in the general region of 9% caused a consistent lowering of flavor score. This effect varied from slight, one point or less, to a moderate two points. In one sense, this hydration effect is an interference in the study of flavor compounds. In another, it may prove useful in producing, under controlled conditions, an off flavor which some judges considered similar to that found in certain types of stale flavor development.

References

(1) Abbot, J., Waite, R., and Hearne, J. F. 1961. Gas Packing Milk Powder with a Mixture of Nitrogen and Hydrogen in the Presence of Palladium Catalyst. J. Dairy Research, 28: 285.

(2) Hanrahan, F. P., Tamsma, A., Fox, K. K., and Pallansch, M. J. 1962. Production and Properties of Spray-Dried Whole Milk Foam. J. Dairy Sci., 45: 27.

(3) Kurtz, F. E. 1965. Direct Recovery of Added Ketones from Foam-Dried Whole Milk. J. Dairy Sci., 43: 269.

(4) Liming, N. E. 1966. Consistency of a Trained Taste Panel. J. Dairy Sci., 49: 628.

(5) Sinnamon, H. I., Aceto, N. C., Eskew, R. K., and Schoppet, E. F. 1957. Dry Whole Milk. I. A New Physical Form. J. Dairy Sci., 40: 1036.

(6) Tamsma, A., Kurtz, F. E., and Pallansch, M. J. Unpublished data.

(7) Tamsma, A., Kurtz, F. E., Rainey, N., and Pallansch, M. J. Unpublished data.